ANNOTATION
dissertation for the degree of Doctor of Philosophy (PhD)
according to the educational program 8D07203 – "Metallurgy"

TUREBEKOVA KARAKAT SERIKOVNA

DEVELOPMENT OF THE TECHNOLOGY FOR THE PRODUCTION OF NON-FERROUS METAL CONCENTRATE AND SILICON OXIDE FROM DUMP TAILINGS OF THE ENRICHMENT PLANTS

Relevance of the dissertation work. This work is aimed at solving the problem of processing barite-containing waste. The problem is due to the lack of technology that allows effectively processing such waste and obtaining marketable products from it.

Stale tailings from the enrichment of polymetallic ores were considered as raw materials that will be processed in the distant future at a higher level of technology and technology. But due to the environmental situation in the regions where these tailings are located, this point of view must be reconsidered.

Long-term storage of sulfide raw materials in the oxidizing conditions of tailings dumps causes irreversible changes, and also leads to environmental pollution and loss of metals.

The problem of processing barite-containing waste with a complex mineralogical composition, which makes the use of traditional processing methods - acid leaching and flotation - ineffective.

Barite-containing waste includes waste tailings from the Karagaily concentrating plant, which are stored in tailings dumps. These tailings ponds occupy a huge area and are sources of soil, air and water pollution with heavy metals and barite dust.

Basis and initial data for developing the topic. Waste from the processing of barite-polymetallic ores from the Karagaily deposit has great economic potential. They contain up to 40% $BaSO_4$; *Cu* - 0.2%; *Zn* - 0.6%; *Pb* - 0.5% according to which these wastes can be classified as industrial polymetallic barite raw materials. They have an advantage over mineral raw materials - they do not require the cost of extraction from the subsoil and grinding. The waste reserves are significant – up to two tens of millions of tons.

The problem of processing this type of raw material is the opening of intergrowths of ore minerals with silicon-containing gangue minerals. A promising solution to the problem is the opening of raw materials using chemical methods and, in particular, the method of ammonium fluorine sulfate desiliconization.

The behavior of the components of barite-containing waste - barite, metal sulfides - during ammonium fluorine sulfate sintering has not been studied. It is necessary to theoretically and experimentally substantiate the effectiveness of using ammonium fluorine sulfate stripping of barite-containing waste.

The work is devoted to finding a solution to the problem of recycling barite-containing waste. Solving this problem is of environmental and economic importance.

The environmental side of the problem is due to the negative impact of barite-containing waste on the environment:

- within the tailings ponds, they occupy significant areas of public land;
- are a source of soil, air and water pollution.

Solving the problem would improve the environmental situation around enterprises producing this type of waste.

From an economic point of view, the disposal of barite-containing waste is necessary, firstly, in terms of reducing the material costs of maintaining tailings dumps. Secondly, a solution to the problem would turn barite-containing waste into an industrial raw material source of barite, silica, and heavy non-ferrous metals, which would differ favorably from conventional mineral deposits in that it is located on the surface and does not require the cost of extraction from the subsoil.

The purpose of the work – development of a combined technology for obtaining a collective concentrate of non-ferrous metals and silicon oxide from the waste tailings of the Karagaily concentration plant.

Object of study is waste from processing barite-polymetallic ores of the Karagaily deposit.

Subject of study: processes of sintering and desiliconization of barite raw materials, consumption of reagents for flotation production of concentrate.

Research objectives. In accordance with this goal, the dissertation sets the following objectives:

- study of the material, granulometric composition and properties of waste tailings from the processing plant;
- thermodynamic analysis of the interaction of raw material components with opening reagents;
- development of a scheme for fluoro-ammonium sulfate opening of raw materials and flotation of waste tailings in laboratory conditions;
- development of a barite product leaching scheme;
- development of a scheme for producing silicon oxide;
- study of flotation of desiliconized product and determination of conditions for obtaining concentrates of non-ferrous metals;
- selection of scheme and reagents for flotation. Determination of optimal flotation conditions using the experimental planning method;
- testing of the developed technology in large-scale laboratory tests.

Scientific novelty consists of a method for processing barite-containing waste - a combination of flotation and chemical (desiliconization) enrichment. Chemical enrichment is based on the use of fluorine and ammonium sulfate salts for opening and desiliconization of barite material with the extraction of silicon oxide into a separate product and obtaining a concentrate of non-ferrous metals.

A review of scientific and technical literature and patent research in this area indicate the novelty of the proposed scientific development.

And also in this work for the first time:

- a thermodynamic analysis of the interaction of raw material components with opening reagents was carried out;
- a mathematical model for opening raw materials in the form of an equation was created and optimal process conditions were determined;
- a mathematical model of the process of silicon extraction from barite raw materials by solution leaching was created;
- a mathematical model of flotation was created in the form of an equation and the optimal process conditions for obtaining a concentrate from a desiliconized product were determined.

Practical value of the work:

- the chemical and granulometric composition of barite raw materials has been studied;
- a thermodynamic analysis of the interaction of raw material components with opening reagents was carried out;
- a scheme for fluoro-ammonium sulfate opening of raw materials has been developed;
- the optimal conditions for aqueous leaching of barite raw materials in the presence of ammonium fluoride were determined: temperature 95°C; time 80 min. and the concentration of the initial ammonium fluoride solution is 25%;
- the conditions for obtaining silicon oxide from barite raw materials by a thermochemical method have been determined: sintering with ammonium bifluoride and ammonium sulfate at temperatures of 200 and 400°C for 3 hours; hydrolysis of captured ammonium hexafluorosilicate with a 10% ammonia solution;
- selected collective scheme and reagents–copper sulfate, potassium butyl xanthate, T-92 for flotation of barite raw materials;
- the optimal conditions for flotation of barite raw materials and obtaining a collective concentrate were determined: consumption of copper sulfate 150 g/t; pulp agitation time with copper sulfate is 5 minutes; xanthate consumption 400 g/t;
- the conditions have been determined and a scheme for obtaining barite concentrate has been developed;
- conditions have been determined and schemes for regeneration of opening reagents have been developed;
- technological regulations have been drawn up for the processing of barite-containing waste using new technology;
- a test report was drawn up for flotation enrichment of barite raw materials after chemical opening on the basis of the Zh. Abishev Chemical-Metallurgical Institute.

Research methods. The composition of barite-containing raw materials was studied using the methods of chemical, mineralogical, sieve and spectral analysis. The presence of silicon oxide up to 41%, barium sulfate up to 33%, zinc, lead and copper sulfides in the amount of up to 2% was established.

A thermodynamic analysis was carried out and the fundamental possibility of selective thermochemical opening of barite raw materials using a combination of ammonium bifluoride and ammonium sulfate in the range of 200 – 400°C was shown.

The sintering of barite raw materials with ammonium sulfate and ammonium bifluoride was studied using the experimental planning method. A mathematical model of the process was obtained and the optimal sintering conditions were determined - temperature 200 - 400°C, ammonium sulfate consumption 77% and ammonium bifluoride consumption 100%.

Using the planning method, experiments on water leaching of barite raw materials in the presence of ammonium fluoride were carried out. A mathematical model of the process was obtained and the optimal conditions for leaching barite raw materials were determined, ensuring the production of a desiliconized concentrate with the composition, mass. %: SiO_2 – 1.9; $BaSO_4$ – 67.3; *Fe* – 8.4; *Zn* – 1.09; *Pb* – 0.91; *Cu* – 0.36.

A thermochemical scheme for producing silicon oxide from barite raw materials has been developed. This method ensures deep desiliconization of raw materials to produce silicon oxide of the "white soot" grade. Research has been carried out on the flotation of barite raw materials. The effectiveness of collective flotation of a desiliconized barite product has been established, which ensures the extraction of copper into a collective concentrate of at least 85%, lead 81% and zinc 80%. At the same time, the quality of barite concentrate increases to 76%. These data are confirmed by a test report carried out at the Zh. Abishev Chemical-Metallurgical Institute.

Using the planning method, experiments were carried out to optimize the conditions for collective flotation of desiliconized barite raw materials. A mathematical model of the process was obtained and optimal flotation conditions were determined, which ensure an increase in the extraction of non-ferrous metals into the collective concentrate by up to 98%.

Provisions for defense:

- results chemical, mineralogical, sieve and spectral analysis of feedstock;
- results of thermodynamic analysis and the fundamental possibility of selective thermochemical opening of barite raw materials;
- results of sintering barite raw materials with ammonium sulfate and bifluoride;
- results of research on flotation of barite raw materials;
- results of experiments to optimize the conditions for collective flotation of desiliconized barite raw materials.

Place of performance of research work.

The work was carried out at the Department of "Nanotechnology and Metallurgy" of Karaganda Technical University, in the laboratory of "Chemistry and Technology of high-silicon materials" of the Chemical and Metallurgical Institute named after Zh. Abishev.

Personal contribution of a doctoral student to writing a dissertation.

The author participated in determining the purpose of the work and setting research objectives, as well as in writing articles and abstracts. Personally, the author obtained the main part of the scientific and practical results of this work, which determines both the scientific novelty and the practical value of the work as a whole. In addition, the entire complex of applied work on the development of schemes for opening raw materials and collective flotation of the desiliconized product, as well as all thermodynamic calculations, was carried out personally and carried out enlarged laboratory tests with obtaining a certificate and drawing up technological regulations.

Approbation of the work.

Based on the results of the research, 8 works were published in domestic and foreign publications, including:

- 2 article in an international scientific journal (“Metalurgija” (Croatia));
- 3 articles recommended by the Committee for Quality Assurance in the Field of Science and Higher Education of the Ministry of Science and Higher Education of the Republic of Kazakhstan (“Proceedings of the University” No. 4 (89) (Karaganda, Kazakhstan), “Mining Journal of Kazakhstan” No. 12 (212), (Almaty, Kazakhstan), “Proceedings of the University” No. 1 (90) (Karaganda, Kazakhstan));
- as well as the research results were presented by the author in 3 reports at international scientific and practical conferences.

The data obtained were confirmed by a test report carried out at the Zh. Abishev Chemical-Metallurgical Institute and the technological regulations were drawn up.

Structure and scope of the dissertation. The dissertation consists of an introduction, a main part of 5 chapters, a conclusion, a list of sources used and 3 appendices. The volume of the dissertation is 120 pages of typewritten text, the work contains 29 figures, 71 tables, a list of used sources, including 84 titles.